

Europalachoa Potantamit

European Patent Office

CONTROL CONTROL CONTROL CONTROL



11 Publication number: 0 649 888 A2

12

EUROPEAN PATENT APPLICATION

(21) Application number: EX307857.5

(5) Int. Ci.<sup>8</sup>: C09D 11/00

2 Date of filing: 26.10.04

@ Priority: 23.10.03 JP 267481/03

63.62.84 JP 11637/64 63.62.84 JP 11632/64 11.63.84 JP 07676/64 24.63.84 JP 182748/84

43 Date of publication of application: 26.04.95 Bullotin 95/17

(A) Designated Contracting States:

CH DE FR GB IT LI ML SE

(1) Applicant: SEIKO EPSON CORPORATION 4-1, Nichlohinjuku 2-chomo Shinjuku-ku Tokyo-eo (JP) (7) Inventor: Yotako, Mocahire elo Salto Escon Consorazion, 3-4, Orro 3-chanto Surro-chi, Nagano-tron (JP)

(14) Representative: Hyte, Otene Blington and Fife Proceed Meuse 8 Penterolo Road Sevenceta, Kont TX18 14R (CB)

- (4) Inta composition for inta jot recording.
- An int. composition for an int. jet recording to provided which has excellent various properties, particularly can realize printing with less bleeding. The Int. composition comprises a dye, propytens glycol monon-butyl either (PGmSE) and/or dipropytens glycol monon-butyl either (DPGmSE) and a water-colubio glycol either other than PGmBE and DPGmBE, the total amount of PGmBE and DPGmBE being 3 to 30% by weight based on the int. composition.

AMOUNT OF ORGANIC SOLVENT ( M1 %)

JPA8-113739

FIG. I.

### BACKGROUND OF THE INVENTION

#### Technical Field

The present invention relates to an ink composition for ink jet recording.

#### Background Art

20

45

40

Vertous properties are required of intercompositions for intercording. For example, intercording for intercording should provide a print on various recording media without bleeding. In order to prevent bleeding, vertous intercording have hitherto been proposed in the art (for example, Japanese Patent Publication Nos. 2807/1990, 15542/1989 and 3837/1990).

Furthermore, a proposal has been made on the prevention of bleeding through an improvement in penetration of an ink composition. For example, U.S. Patent No. 9,169,075 discloses an ink composition containing disthylene glycol monobutyl either. U.S. Patent No. 9,163,502 discloses an ink composition containing a curfactant. U.S. Patent No. 5,198,056 discloses an ink composition containing disthylene glycol monobutyl either and a surfactant. Disthylene glycol monobutyl either is called "butylcarbitol" and disclosed in, for example, U.S. Patent No. 3,291,580. U.S. Patent No. 2,083,372 discloses an ink composition containing disthylene glycol either.

According to the above ink compositions, although bleading can be prevented to some extent, an ink composition, which is less likely to cause bleeding, has been decired in the crt.

In recent years, recycled paper has become used from the viewpoint of environmental protection. The recycled paper comprises components of various types of paper. Thus, penetration of an ink composition into the recycled paper varies from component to component, which results in bleeding in the print. In order to prevent bleeding in the recycled paper, a method has been proposed wherein recording paper is heated at the time of printing. In this method, however, a cartain period of time is required for the paper to be heated to a predetermined temperature. Furthermore, power consumption becomes high, which is not cost-offsetive. Moreover, there is a fear of the recording paper and print being detarlorated by heating.

The ink composition for ink jet recording should satisfy further property requirements including that it should provide a good print density and a long intermittent printing time and be stable and less likely to clogging of nozzles.

### SUMMARY OF THE INVENTION

Accordingly, an object of the present invention is to provide an ink composition for ink jet recording which has various excellent properties, especially an ink composition for ink jet recording which can realize printing with less bleeding.

The ink composition for ink jet recording according to the present invention comprises a dye, propylene glycol mono-n-butyl ether (PGmBE) and/or dipropylene glycol mono-n-butyl ether (PGmBE) and a water-coluble glycol other other than PGmBE and DPGmBE, the amount of PGmBE and DPGmBE being 3 to 30% by weight based on the ink composition.

### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a graph showing the penatration times of ink compositions wherein O represents the penatration times of ink compositions comprising the same components as the ink composition of Example A1 except that the emounts of disthylene glycol monosthyl ether and 1,4-butanedial added as organic solvents were varied in the range of from 2 to 35% with the ratio of disthylene glycol monosthyl other to 1,4-butanedial being maintained at 2:1; 0, 0 and A represent the penatration times of ink compositions having the comprising the same components as the ink composition of Example A1 except that 10% Surfynol 485, 10% disthylene glycol mono-n-othyl ether and 1% Surfynol 485 were used instead of 10% PGmBE. respectively, and the amounts of disthylene glycol monosthyl ether and 1,4-butanedial added were varied with the ratio of disthylene glycol monosthyl ether to 1,4-butanedial added were varied with

Fig. 2 is a graph showing the ponatration times of ink compositions wherein  $\Box$  represents the penatration times of ink compositions comprising the came composition as the ink composition of Example A1 except that the amounts of distinglers glycol monosthyl other and PGm8E added were varied with the ratio of distinglers glycol monosthyl either to PGm8E being maintained at 1:1 and O represents the penatration times of ink compositions comprising the same components as the ink composition of Example A1 except

that DPGmBE was used instead of PGmBE;

Fig. 3 is a graph showing OD values of prints obtained using ink compositions comprising the same components as the ink composition of Example B1 except that the amount of ures was varied; and Fig. 4 is a graph showing the intermittent printing times of ink compositions comprising the same components so the ink composition of Example D1 except that the thiodighycol concentration was varied.

### DETAILED DESCRIPTION OF THE INVENTION

10

19

25

20

20

The ink composition of the present invention comprises PGmBE and/or DPGmBE in a total amount of 3 to 30% by weight, preferably 5 to 10% by weight, based on the ink composition. The content of PGmBE and DPGmBE within the range can effectively prevent bleeding of the ink on recording paper.

The ink composition of the present invention may comprise a water-caluble glycol either in addition to PGm8E and/or DPGm8E.

Without intending to be bound by the following theory, it is believed that a combination of PGmBE end/or DPGmBE with a water-soluble glycol ethar enables the above amount of PGmBE and DPGmBE to be resilized. PGmBE and DPGmBE has only a limited solubility in water. According to studies of the present inventors, for example, when PGmBE and DPGmBE are used alone, the calubility thereof in water is 5 to 6%, whereas the presence of other components of the ink composition, cuch as dyes, lowers the solubility to less than about 2%. However, the addition of a water-soluble glycol ethar to the system enables the above amount of PGmBE and DPGmBE to be realized. In addition, various properties of the ink composition can be improved. More apocifically, for example, prints free from bleeding can be provided.

Proferred stramples of the tester-activitic glycol include ethylene glycol monosityl ethers, tristhylene glycol monosityl ethers, tristhylene glycol monosityl ethers, propylene glycol monosityl ethers and alkoxy-substituted aliphatic alcohola. Still preferred examples thereof include ethylene glycol mono-C1\_calkyl ethers, diethylene glycol mono-C1\_calkyl ethers, tristhylene glycol mono-C1\_calkyl ethers, tristhylene glycol mono-C1\_calkyl ethers, propylene glycol mono-C1\_calkyl ethers, dipropylene glycol mono-C1\_calkyl ethers and C1\_calkoxy-substituted C1\_caliphatic alcohola. Specific examples of the tester-caluble glycol include ethylene glycol monomathyl ether, ethylene glycol monomathyl ether, ethylene glycol monomathyl ether, ethylene glycol monomathyl ether, diethylene glycol monomathyl ether, diethylene glycol mono-n-butyl ether, ethylene glycol mono-iso-propyl ether, ethylene glycol mono-t-butyl ether, diethylene glycol mono-t-butyl ether, diethylene glycol mono-t-butyl ether, propylene glycol mono-t-butyl ether, dipropylene glycol mono-teleo-propyl ether and dipropylene glycol mono-iso-propyl ether.

The water-soluble glycol other may be added in any amount so far as the ink penetration rate is sufficient and PGmBE and DPGmBE are not subjected to phase expanation. The amount of the water-soluble glycol other added, however, is preferably in the range of from 5 to 60% by weight, still preferably in the range of from 5 to 15% by weight, based on the ink composition.

Further, according to a preferred embodiment of the present invention, the websr-coluble glycol ether is preferably added in an amount of not less than 0.5% by weight, still preferably 0.8 to 3% by weight, based on the total amount of PCmBE and DPCmBE. The amount of the websr-coluble glycol other within the range improves the dissolution stability of PGmBE and DPCmBE. At the same time, it is possible to prevent the occurrence of such a phenomenon that part of cil-coluble components contained in the ink composition bleads on the paripherty of a print on a recording medium. Furthermore, the oder of glycol other can be reduced.

According to a further preferred embediment of the present invention, the ink composition of the present invention may contain uses and/or a uses derivative. The addition of the uses and/or uses derivative can curprisingly improve print density and prevent clogging of ink delivery parts. Examples of the uses derivative include athyleneures, thiouses, blures, bluret or tetramethyluses. The amount of the uses and/or uses derivative added can be freely determined so far as the print density of the ink composition can be improved and, at the same time, clogging of nozzles can be effectively prevented. It, however, is preferably in the range of from 2 to 20% by weight, still preferably in the range of from 5 to 10% by weight, bacad on the ink composition.

According to a further preferred embediment of the present invention, the ink composition of the present Invention may further comprise thioglycol. The addition of thioglycol surprisingly enables the intermittent printing time to be protonged. The term "intermittent printing time" used herein is intended to mean the period of time for which the ink can remain undelivered without causing any unfeverable phenomena, such as clogging. In the subsequent resumption of printing. In ink jet recording printers, the ink is delivered at given time intervals

#### FP 0 649 823 AZ

10

for the purpose of preventing nozzles from being clogged due to drying of the vicinity of the nozzles. This operation is generally called "flushing." The ink composition having a long intermittent printing time can prolong the time interval for flushing. This can advantageously avoid wasting the ink composition and, at the earns time, increase the printing speed. Moreover, the addition of this glycol is advantageous also from the viewpoint of improving the stability of the ink composition.

According to a further preferred embediment of the present invention, the ink compaction of the present invention may further comprise a surfactant. The addition of the surfactant may improve proporties of the ink composition, such so the intermittent printing time. Preferred examples of the surfactant include anionic curfactants, amphoteric surfactants, cationic curfactants and nonionic curfactants. Examples of the anionic curfactant include alkylsulfocarbonates, o-clefineutfonates, polyckycthylane alkyl ether occiates, N-ocylomino acid and ito saits, N-acytmethyltaurine saits, alkylculfate polyoxyolkyl ether sulfates, alkylculfate polyoxyethylone other phoophetes, rooin soop, cestor oil sulfates, leuryl elcohol sulfetes, elkylphenol phoephetes, elkyl phosphatos, etkylollyleutionates, diothylsulfocuccinetes, diothylhoxyleutfocuccinetes and dioctylsulfocuccinates. Examples of the cetionic surfectant include 2-viny/pyridine derivestives and poly(4-viny/pyridine) derivetives. Examples of the emphoteric curfactant include issuryidimathylaminoscatic sold betains, 2-alkyl-N-cerboxymathyl-N-hydroxyathylimidazolinium bataina, coconut oil fatty acid amide propyldimathylaminoacatic acid bataina, polyoctylpolyeminoathylghycina and othar imidazolina derivetives. Examples of the nonlante curfactant include ether nonionic curfactante such as polyoxyethylane nonylphenyl ether, polyoxyethylane octylphenol ether, polyoxysthylene dodscylphenyl ether, polyoxysthylene alkylellyl ether, polyoxysthylene disyl other, polyoxyethylens leuryl ether, polyoxyethylene elkyl ethers and polyoxyelkylene alkyl ether, ester nonionic curriectante cuch as polyoxycithylencoleic acid, polyoxycthylencoleic acid ectars, polyoxycthylencoleicaric acid esters, corbitan teurate, sorbitan monostearate, sorbitan monocleate, sorbitan sesquioleate, polyonyemylene monopleate and polyenyethylene sterrate; acetyl glycol nontonic surfactants such as 2,4,7,8-tatramethyl-5-decyn-4,7-dial, 3,6-dimethyl-4-octyn-3,6-dial, 3,5-dimethyl-1-haxyn-3-cl, for example. Surfynol 104. 82, 465, and TG manufactured by Nisshin Chamical Industry Co., Ltd.; and fluorosurfactants such as fluoringsubstituted altry exters and parflucrosity/carbonylates, for example, Plangent series manufactured by Nece Co., Ltd., Lodyna parties manufactured by Chiba-Golgy Limbod, Japan, Zonyi portae manufactured by Du Pont Kabuchiki Kalaha, Monflor saries manufactured by ICI, Surfluon series manufactured by Asahi Glass Co., Ltd., Unidyna saries manufactured by Daikin Industrias, Ltd. and FC scrips manufactured by Sumitomo 3M Ltd. Fluorosurfectants, particularly amphotetic or nontonic surfactants are especially preferably used. Wittle the amount of the surfactant added may be freely determined, for the fluoresurfactant, the amount is preferably about 1 to 10,000 ppm.

According to a further preferred embodiment, the ink composition of the present invention may further comprise a water-coluble organic selvant. Examples of the water-coluble organic selvant include cityl elected having 1 to 4 extron atoms such as othered, methand, butanol, propanel and isopropanel; polyhydric electeds or glycols such as ethylene glycol, disthylene glycol, thathylene glycol, polyethylene glycol having a molecular weight of not more than 600, propylene glycol, dipropylene glycol, tripropylene glycol, polypropylene glycol having a molecular weight of not more than 400, 1,3-butylene glycol, 1,3-propenedial, 1,4-butenedial, 1,5-pentenedial, 1,8-hexanedial, thiodiglycol, glycorin, meso-erythribal and pentacrythribal; formsmide, acctamide; dimethyleuffoxide; corbital; corbital; corbital; corbital; corbital; corbital; corbital;

Further, the ink composition of the present invention may contain various additives for the purpose of improving various properties of the ink composition. Examples of the additive include enthaptic agents, milder-proofing agents, pH adjustors and viscosity modifiers. Specific examples of the enthaptic and milder proofing agents include addition benzoats, addition partiachlorophenol, addition 2-pyridinethici-1-oxide, addition acrobicate, addition and 1,2-dibenzothlezalin-3-one (Proxel CRL, Proxel BDN, Proxel GXL, Proxel XL-2 and Proxel TN manufactured by ICI).

Examples of the pH adjustor include amines such a disthenolamine, thethenolamine, propendemine and morpholine; inorganic calts such as possessium hydroxide, codium hydroxide and lithium hydroxide; ammonium hydroxide; quaternary ammonium hydroxide such as tetramethylammonium; carbonates and phoophates such as possessium carbonate, sodium carbonate and lithium carbonate; W-methyl-2-pyrrotidone; ureas such as urea, ticurea and tetramethyluma; allophanetes such as allophanete and methyl allophanete; and blurete such as bluret, dimethylbiuret and tetramethylbiuret.

Exemples of the viscosity adjustor include polyvinyl alcohol, hydroxypropyl cellulese, hydroxyethyl cellulose, mathyl cellulose, polyacrylates, polyarylates, polyarylates

The edicrente usable in the ink composition of the present invention include, but not limited to, direct dyes, acid dyes, basic dyes, reactive dyes and food dyes.

Specific preferred examples of the direct dys include C.I. Direct Black 2, 4, 8, 11, 14, 17, 19, 22, 27, 32, 36, 41, 48, 51, 58, 62, 71, 74, 75, 77, 78, 80, 105, 106, 107, 108, 112, 113, 117, 132, 148, 154, 168, 171 and

#### EP 0 8≤19 8≤30 △2

184, C.I. Direct Yellow 1, 2, 4, 8, 11, 12, 24, 26, 27, 28, 33, 34, 39, 41, 42, 44, 48, 50, 51, 58, 72, 65, 66, 97, 88, 83, 100, 110, 127, 135, 141, 142 and 144, C.I. Direct Orenge 6, 8, 10, 23, 29, 39, 41, 49, 51, 82 and 102, C.I. Direct Red 1, 2, 4, 8, 9, 11, 13, 15, 17, 20, 23, 24, 28, 31, 33, 37, 39, 44, 48, 47, 48, 51, 59, 62, 63, 73, 75, 77, 79, 80, 81, 83, 84, 85, 87, 89, 90, 84, 95, 99, 101, 108, 110, 145, 189, 197, 224, 225, 226, 227, 230, 250, 268 and 257, C.I. Direct Viciet 1, 7, 9, 12, 35, 48, 51, 80 and 94, C.I. Direct Blue 1, 2, 8, 8, 12, 15, 22, 25, 34, 69, 70, 71, 72, 75, 76, 78, 80, 81, 82, 83, 86, 97, 90, 88, 103, 108, 110, 120, 123, 158, 163, 165, 192, 193, 194, 195, 193, 199, 200, 201, 202, 203, 207, 218, 236, 237, 239, 246, 256 and 287, C.I. Direct Green 1, 6, 6, 28, 33, 37, 83 and 64, and C.I. Direct Brown 1A, 2, 6, 23, 27, 44, 58, 85, 100, 101, 106, 112, 173, 184, 195, 209, 210 and 211.

Examples of the acid dye include C.I. Acid Black 1, 2, 7, 16, 17, 24, 26, 28, 31, 41, 48, 52, 58, 60, 63, 84, 107, 109, 112, 118, 119, 121, 122, 131, 155 and 158, C.I. Acid Yellow 1, 3, 4, 7, 11, 12, 13, 14, 17, 18, 19, 23, 25, 29, 34, 36, 38, 40, 41, 42, 44, 49, 53, 55, 59, 61, 71, 72, 78, 78, 79, 99, 111, 114, 116, 122, 135, 142, 181 and 172, C.I. Acid Crango 7, 8, 10, 19, 20, 24, 28, 33, 41, 45, 51, 56 and 64, C.I. Acid Red 1, 4, 6, 8, 13, 14, 15, 18, 19, 21, 26, 27, 30, 32, 34, 35, 37, 40, 42, 44, 51, 52, 54, 57, 80, 82, 63, 85, 87, 88, 89, 92, 94, 97, 103, 108, 110, 111, 114, 115, 119, 129, 131, 134, 135, 143, 144, 152, 154, 155, 172, 176, 180, 184, 188, 187, 249, 254, 258, 289, 317 and 318, C.I. Acid Victet 7, 11, 15, 34, 35, 41, 43, 49, 51 and 75, C.I. Acid Blue 1, 7, 9, 15, 22, 23, 25, 27, 29, 40, 41, 43, 45, 51, 53, 55, 58, 59, 62, 78, 80, 81, 83, 80, 92, 93, 102, 104, 111, 113, 117, 120, 124, 128, 138, 145, 187, 171, 175, 183, 229, 234, 238 and 249, C.I. Acid Green 3, 9, 12, 18, 19, 20, 25, 27, 41 and 44, and C.I. Acid Brown 4 and 14.

Examples of the basic dye include C.I. Seelc Black 2 and 8, C.I. Seelc Yellow 1, 2, 11, 14, 21, 32 and 38, C.I. Seelc Orange 2, 15, 21 and 22, C.I. Seelc Red 1, 2, 9, 12, 13 and 37, C.I. Seelc Violet 1, 3, 7, 10 and 14, C.I. Seelc Blue 1, 3, 5, 7, 9, 24, 25, 26, 28 and 29, C.I. Seelc Green 1 and 4, and C.I. Seelc Brown 1 and 12.

Examples of the reactive dye include C.J. Reactive Black 1, 3, 5, 8, 8, 12 and 14, C.I. Reactive Yellow 1, 2, 3, 12, 13, 14, 15 and 17, C.I. Reactive Orange 2, 5, 7, 16, 20 and 24, C.I. Reactive Red 6, 7, 11, 12, 15, 17, 21, 23, 24, 35, 38, 42, 63, 68, 84 and 184, C.I. Reactive Violet 2, 4, 5, 8 and 9, C.I. Reactive Blue 2, 5, 7, 12, 13, 14, 16, 17, 18, 19, 20, 21, 25, 27, 28, 37, 38, 40 and 41, C.I. Reactive Green 5 and 7, and C.I. Reactive Brown 1, 7 and 16.

Examples of the food dye include C.I. Food Black 1 and 2, C.I. Food Yellow 3, 4 and 5, C.I. Food Red 2, 3, 7, 9, 14, 52, 87, 92, 94, 102, 104, 105 and 103, C.I. Food Violet 2, C.I. Food Blue 1 and 2, and C.I. Food Green 2 and 3.

Further, it is also possible to utilize Kayaset Black 008A, Direct Deep Black XA and Direct Special Black AXN manufactured by Nippon Kayaku Co., Ltd., Special Black SP Liquid, Bayecript Black SP Liquid, Levecell Turqueise Blue KS-6GLL and Pyranine menufactured by Bayer Japan Ltd., Jl. 8K-2 and Jl. 8K-3 manufactured by Sumitomo Chemical Co., Ltd., JPK-81L, JPX-127L, JPK-139 and C.I. Fluorescent Brightening Agent 14, 22, 24, 32, 84, 85, 86, 87, 90, 134, 166, 167, 169, 175, 176 and 177 manufactured by Ortant Chemical Industries, Ltd.

Various properties of the ink composition according to the present invention can be properly determined so as to be suitable for ink jet recording. In this context, it is preferred to take into consideration particularly the feaming property, surface tension and forward contact angle thereof with nozzle face of an ink jet recording hoad. More specifically, the feamability of the introompecition as measured at 15°C by the Ress-Miles test is not more than 200 mm as the initial value and not more than 100 mm 5 min after the initiation of the test. The Ross-Miles test is a foarmability test wherein 200 mil of a test colution is fallen down from a height of 90 cm through a small tube of 29 mm inner diameter into a graduated tube of 50mm inner diameter containing 50 ml of the test solution to distermine the height of the resultant fears. The initial value and the value 5 min after the initiation of the test within the above range can prevent the occurrence of foam in an ink passage and an recording head. Thus, the ink composition can be easily filled into nozzles, which enables dropout and other unfavorable phonomens to be effectively prevented. The curface tension of the int composition is preferably in the range of from 15 to 40 mR/m. When the currece tension falls within the above range, the ink composition can be easily filled into nozzica. Moreover, at the carre time, a print having a good quality can be provided. The context angle of the init composition to the nozzle plate of the recording head is preferably in the range. of from 10 to 50°. When the contect angle falls within the range, the int composition can homogeneously wet nozzle faces, co that a print having a good quality can be provided. Further, this provides an advantage that the intermittant printing time can be prolonged.

The ink composition of the present invention can be produced by the conventional method. Specifically, the above components are sufficiently stirred and mixed together, optionally filtered to remove solid matter, thereby preparing an ink composition.

#### EP 0 849 888 AZ

### EXAMPLES

The precent invention will now be described in more datail with reference to the following examples, though it is not limited to these examples only.

In the following examples, printing was carried out using an ink jet printer MJ-500 manufactured by Selko Epcon Corporation, unless otherwise noted.

Furthermore, "%" is by weight unless otherwise noted. Moreover, the ink composition other than those of Example A and Comparative Example A contain 0.1 to 1% of Proxel XL-2 as corresion inhibitor and 0.001 to 0.005% of benzotriazale for preventing the corresion of recording head members.

### Exemple A

10

220

Into compositions of Examples A1 to A8 and Comparative Examples A1 to A3 were prepared by the conventional method. Specifically, the following ingredients were stirred and 5 mixed together, and the mixture was filtered to prepare an intocomposition.

### Exemple A1

C.I. Direct Black 19	2%
PGmBE	10%
Disthylane glycol monoathyl ether	10%
1,4-Busus-4,1	5%
Sodium diectyisuiioouccinate	1.6%
₩atar	Balance

### Example A2

C.I. Food Black 19	2.5%
PGm8E	10%
Diethylena glycol mono-n-butyl ether	10%
Dipropylena glycol	5%
Proxol GXL	0.3%
Watar	Balanca

### Exemple A3

C.J. Actd Graen 9	2.5%
P@mBE	10%
1-Methyl-1-mathoxy butand	15%
Distriylens glycol	5%
Wester	Belenco

### EP 0 840 000 AZ

## Exemple A4

Special Black SP liquid 20%

DPGmBE 10%

Propylene glycol monoathyl ethar 10%

1,3-Dimethyl-2-imidazolidinona 6%

Weter Belance

## Example A5

Direct Special Black ATM 4%
PGmBE 9%
Dipropylens glycol monomethyl ether 12%
N-Methyl pyrrolidene 5%
Water Belance

### Example A6

C.I. Reactive Red 6 4%
PGmBE 9%
DPGmBE 5%

Disthylene glycol mono-n-butyl ather 15%
N-Acyl methyl taurine sodium 1.5%
Water Salance

## 6 Example A7

•	1 1
C.I. Beaic Yellow 11	5%.
PGMBE	10%
Tricthylone glycal monobutyl other	12%
Glycartn	10%
Surfynol 485	1%
Surfynol 104	0.5%
Water:	Selance

## EP 0 (xis 883) A2

### Example A6

C.I. Direct Green 1 3%

C.I. Direct Green 28 1%

PGmBE 10%

Distingions glycol mono-t-butyl other 5%

Distingions glycol mono-n-butyl ether 5%

Distingions glycol 10%

Propytano glycol monomathyl other 8elanco

## Comparative Example A1

C.I. Direct Green 1	3%
Diathylana glycol mono-n-butyl ether	5%
Tetraethylone glycol	16%
Potassium hydroxida	0.1%
₩atar	eonsie B

## Comparative Example A2

20

85

C.I.Diroct Red 227	2.5%
Distingens glycol mono-n-butyl ether	15%
Triathylana glycol	5%
Glycerin	10%
Water	Betance

## Comparative Example A3

C.I. Acid Red 254	2.5%
Diathylona glycol mono-n-butyl ether	7%
Diathylena glycci	15%
Websr	Balanca

### Evaluation Tool A1

The above ink compositions were used to carry out printing on various types of recording paper specified in Table 1. The prints thus obtained were evaluated as follows.

## Blooding

The circularity of dots was evaluated according to the following criteria as a measure of penetration of the ink wherein uneven penetration of the ink gave rise to a lowering in circularity of the dots.

- 5 (i): Very good circularity;
  - O: Slightly lowered circularity which causes no problem for practicel use;
  - A: Lowered circularity which causes a problem for precticel use; and
  - X: Remarkably lowered circularity which renders the ink unsultable for practical use.

### 10 Feethering

20

20

Unaven penatration of an ink along paper fiber, i.e., feathering, was evaluated according to the following criteria.

- (i): Little or no feetharing with very even panatration;
- O: Slight feathering which causes no problem for practical use;
- A: Many leathering which causes a problem for practical user and
- X: Remarkable feathering which renders the influenciable for practical use.

The recults were as given in the following Table 1.

## Table 1

Test item	Type of paper				Ex.	A 					omp	
		1	2	3	4	5	6	7	8	1	2	3
	CONQUEROR Paper	0	0	9	0	0	<b>6</b>	<b>@</b>	<b>9</b>	0	0	0
	FAVORIT X Paper	0	0	0	0	0	0	0	ම	Δ	0	0
	MODO COPY Paper	0	0	0	0	0	0	0	0	я	Δ	х
	RAPID COPY Paper	0	<b>®</b>	0	0	0	<b>Ø</b>	•	<b>©</b>	A	0	0
	EPSOM EPP Paper	0	0	0	(O)	0	0	<b>9</b>	. @	Ħ	A	я
Bleed-	XEROX P Paper	6	<b>ø</b>	0	0	<b>®</b>	0	0	0	Ħ	Δ	ĸ
ing	XEROX 4024 Paper	0	0	<b>Ø</b>	Ф	0	0	8	0	Δ	0	A
	XEROX 10 Paper	0	0	0	0	0	0	0	0	Δ	Δ	Δ
	NEENACH BOND Paper	9	0	0	0	.0	0	0	0	ж	Δ	Δ
	RICOPY 6200 Paper	0	0	0	9	0	0	0	0	Δ	Δ	Δ
	Yamayuri Paper	0	9	0	€	0	0	0	0	ж	×	. ×
	XEROX R Paper	· (9)	9	0	9	0	0	0	0	я	×	ж
	CONQUEROR Paper	9	0	<b>9</b>	0	0	0	0	0	я	Δ	я
	FAVORIT X Paper	9	0	0	0	9	9	0	0	Δ	Δ	Δ
	MODO COPY Paper	9	0	0	0	0	0	0	0	À	Δ	Δ
	RAPID COPY Paper	•	0	0	0	0	0	0	0	я	Δ	Δ
Feath- ring	XEROX P Paper	9	©	0	0	<b>(</b>	0	0	0	×	Δ	×
	XEROX 4024 Paper	9	0	<b>©</b>	9	0	@	0	0	ж	ж	R
•	RICOPY 6200 Paper	0	0	0	9	9	0	0	0	ж	Δ	Δ
	Yamayuri Paper	0	9	0	၁	0	C	င	0	×	*	×
	XEROX R Paper	0	0	Ö	(9)	0	@	0	0	×	ж	н

The recording papers specified in the table are available from the following companies.

#### EP 0 849 838 A2

CONQUEROR Paper. Arjo Wigging Co., Ltd.
FAVORIT X Paper. Favorit Co., Ltd.
MODO COPY Paper. Mode Co., Ltd.
RAPID COPY Paper. Igepa Co., Ltd.
SEPSON EPP Paper. Seiko Epson Corporation
XEROX P Paper. Fuji Xerox Co., Ltd.
XEROX 4024 Paper. Xerox Corp.
XEROX 10 Paper. Xerox Corp.
NEENACH BOND Paper. Kimberly-Clark Co., Ltd.
Yomeyuri Paper. Honshu Paper Co., Ltd.
XEROX R Paper. Xerox Corp.

### Eveluation Tool A2

15

In the introduction prepared in Example A1, the amounts of disthylene glycol monosthyl ethat and 1,4-butanedial added as organic solvents were varied in the range of from 2 to 35% (an increase or a decrease in the amount of the organic solvents was belianced by water) with the ratio of disthylene glycol monosthyl ether to 1,4-butanedial being maintained at 2:1. One  $(1) \mu$  of the introduction was dropped in a spot form on DERSCHER paper. The time tetran for drying the introduction was measured as a penciration time. The results are indicated as O in Fig. 1.

Further, in the int composition prepared in Example AI, 10% Surfynd 465 was used instead of 10% PGmBE, and the amounts of distinglens glycol monosthyl either and 1,4-butanedial added as organic colvents were varied with the ratio of distinglens glycol monosthyl either to 1.4-butanedial being maintained at 2:1. The penstration time of the int composition was measured in the same manner as described above. The results are indicated so 0 in Fig. 1. Moreover, in the int composition prepared in Example A1, 10% distinglens glycol mono-n-butyl other and 1% Surfynol 485 were used instead of 10% PGmBE, respectively, and the amounts of distinglens glycol monosthyl either and 1,4-butanedial added as organic solvents were varied with the ratio of distinglens glycol monosthyl either to 1,4-butanedial being maintained at 2:1. The penatration time of the link composition was measured in the same manner as described above. The results are indicated as  $\Box$  and  $\Delta$  in Fig. 1.

### Evaluation Test A3

In the introduction prepared in Example A1, the amounts of PGmBE and distriptions glycol monosthyl either added were varied with the ratio of PGmBE to distriptions glycol monosthyl either being maintained at 1: 1. The panetration time of the introduction was measured in the same manner as in Evaluation test A2. The results are indicated as  $\Box$  in Fig. 2.

Further, the panetration time of the ink composition was measured in the same manner as described above, except that DPGmBE was used instead of PGmBE. The results are indicated as 0 in Fig. 2.

### Example 8

Int compositions of Examples B1 to B8 and Comparative Examples B1 to B3 were prepared by the conventional method.

## EP 0 600 600 AZ

## Example 81

C.I. Direct Black 19 275 PGm8E 10% Disthylana glycol monoathyl ether 10% 1.4-Butenediol 5% Urea 5% Dipropylema glycol 5% **Water** Balanco

## Exemple 82

10

	C.I. Food Black 2	. 2.5%
20	PGmBE	10%
	Diethylene glycol mono-n-butyl ether	10%
	Dipropylene glycol	<b>5</b> %
<b>2</b> 5 ·		•
	Urge	<b>10</b> 8
	Pronel GML	0.3\$
	#ater	Balance

## **Example 83**

C.J. Acid Green 9	2.5%
PGmBE	10%
1-Methyl-1-mathoxy butanol	15%
Uma	7%
Distriylens glycol	5%
Welci	Belance

Balance

## Example 84

Spacial Black SP Hould	20%
DPGmBE	10%
Propylone glycol mencothyl other	10%
enonibilosabimi-2-imidazolidinone	<b>5</b> %
Thiourea	<b>5</b> %
Water	Belanca

## EP 0 €49 833 A2

## Example 85

Direct Special Black AXN	4%
PGmBE	- 9%
Dipropylena glycol monomethyl ether	12%
N-Methyl pyrrolidone	5%
Ethyleneuree	5%
Weter	Belanca

## rs Example 86

20

C.I. Reactive Red 8	4%
PGmBE	8%
DPGmBE	5%
Distinyions glycol mono-n-butyl ether	16%
N-Acylmathyltaurina sodium	1.5%
Blurea	3%
Water	Balanca

## Example B7

	C.I. Basic Yellow 11	. 5%
	PGmBE	108
25	Triethylene glycol monobutyl ether	128
	Glycerin	108

40		Surfynol	465					18
		Surfynol	104	٠,	•		٠.	0.58
	<	Biuret				• .		3&
45		Water						Balance

**£0** 

### EP 0 849 830 A2

### Example 68

C.I. Direct Green 1	3%
C.I. Direct Green 28	1%
PGmBE	10%
Disthylens glycol mono-t-butyl ether	5%
Disthylens glycol mono-n-butyl ether	5%
Distinyions glycol	10%
Tetramathylurea	3.5%
Propylane glycol monomathyl ether	Balanca

### Comparativo Example 81

ы		

10

C.I. Direct Grean B	3%
C.I. Direct Green 1	1%
Diathylona glycol mono-n-butyl ether	5%
Tetraethylene glycol	15%
Potacetum hydroxida	0.1%
Weber	Satanca

20

26

## Comparativo Exemple 82

2	ŗ	ă		

C.I.Direct Red 227	2.5%
Diathylena glycol mono-n-butyl ether	15%
Triothylono glycal	5%
Glycerin	10%
Water	නොක්ෂ <b>ි</b>

## Comparativo Example 83

35

C.I. Acid Red 264	2.5%
Distrigions glycol mono-n-butyl ether	7%
Diathylena glycol	15%
Water	Balanca

**...** 

## **Evaluation Test B1**

The above ink compositions were used to carry out printing on various types of recording paper specified in Table 2. The printe thus obtained were evaluated in the same menner as was described in Evaluation Test

# ED 0 870 030 73

A1

The recults were as given in Table 2.

## EP 0 649 690 AZ

Table 2

Test item	Type of paper				Ex.	В					omp	
		1	2	3	4	5	6	7	8	. 1	2	3
	COMQUEROR Paper	<b>0</b>	<b>Ø</b>	0	0	0	0	<b>®</b>	<b>@</b>	0	<b>®</b>	C
	FAVORIT X Paper	<b>ø</b>	0	0	0	0	0	0	9	<b>A</b>	0	c
	MODO COFY Paper	0	0	0	0	0	<b>9</b>	<b>ø</b>	<b>ø</b>	ж	Δ	R
	RAPID COPY Paper	<b>6</b>	<b>©</b>	0	0	0	Ø	0	<b>ø</b>	A	0	c
,	EPSON EPP Paper	<b>6</b>	0	0	0	0	Ø	<b>@</b>	<b>(4)</b>	×	A	я
Bleed-	XEROX P Paper	<b>©</b>	0	0	0	0	0	0	0	x	<u>A</u>	×
ing	XEROX 4024 Paper	Ø	0	0	0	0	Ø	ூ	Ø	Δ	0	Δ
	XEROX 10 Paper	9	0	0	0	<b>0</b>	<b>9</b>	<b>Ø</b>	0	Δ	Δ	Δ
	NEENACH BOND Paper	9	0	0	0	0	0	9	ø	ж	Δ	Δ
_	RICOPY 6200 Paper	Ð	9	0	0	0	0	0	•	Δ	Δ	۵
	Yamayuri Paper	0	0	0	0	0	9	9	0	ж	ж-	×
·	KEROX R Paper	Ð	0	0	0	0	9	9	0	и	ж	×
	COMQUEROR Paper	0	0	0	0	0	9	0	9	ж	Δ	×
	FAVORIT X Paper	<b>9</b>	9	9	0	6	0	0	0	Δ	Δ	△
	MODO COPY Paper	0	0	0	0	0	9	e	0	Δ	Δ	Δ
	RAPID COPY Paper	9	0	0	0	0	0	0	0	×	Δ	Δ
Feath- ring	XEROX P Paper	0	0	0	0	0	0	0	0	×	Δ	ж
	XEROX 4024 Paper	0	0	0	0	0	0	0	@	ж	я	×
	RICOPY 6200 Paper	0	9	0	<b>9</b>	0	0	0	0	×	۵.	△
	Yamayuri Paper	0	0	0	0	0	Ö	0	0	ж	ж	я
	XEROX R Paper	0	0	0	9	0	0	0	0	×	×	я

**5**5

### **Evaluation Test 82**

The OD values of the prints obtained using the ink compositions of Examples B1 to B8 were measured. Separately, ink compositions were prepared in the same manner as in Examples B1 to B8, except that neither ures nor a ures darivative was added. The OD values of the prints obtained using the ink compositions were measured. The OD ratio was determined by the following formula:

OD ratio = (A/B) x 100 - 100

wherein

10

15

. 20

A is OD value when ures or a ures modification product was added, and B is OD value when neither ures nor a ures modification product was added. The OD ratios were as follows.

Examples	CD ratio			
1	8.8			
2	9.7			
3	7.7			
4	9.2			
5	6.5			
8	2.3			
7	3.2			
8	1.5			

### Evaluation Test 83

In the ink composition of Example B1, the amount of urea added was varied. A change in the total amount of the composition caused by the increase or decrease in the amount of urea add was balanced by water. The OD values of prints obtained using the ink compositions were measured. The results were as shown in Fig. 3.

### Example C

Inx compositions of Exemples C1 to C8 and inx compositions of Componentive Exemples C1 to C3 were prepared by the conventional mathed.

### Example C1

C.I. Direct black 18	2%
PGmBE	10%
Triathylana glycol monobutyl ether	10%
1,4-Butanediol	2.5%
1,6-Howenedial	2.5%
2-Pyrrolidona	1.5%
Water	Balanca

## EP 0 649 833 AZ

## Example C2

C.I. Food Black 2	2.5%
PGmBE .	10%
Diathylena glycol mono-n-butyl ether	10%
Dipropylena glycol	3%
Tripropylene glycci	3%
Uroa	5%
Water	eanete B

Example C3

15

20

C.I. Acid Green 9	2.5%
PCmBE	10%
1-Methyl-1-mathoxy butand	16%
Propylenc glycol	5%
Triothylane gl <del>ycal</del>	5%
1,5-Pentenedici	5%
Weter	Balanca

Ехетрю С4

Special Black SP liquid	20%
DPGm8E	10%
Propylono glycel meneothyl ether	10%
1,6-Hemanediel	2%
Tripropylene glycol	3%
1,3-Dimethyl-2-tmldazolidinone	5%
Water	නොක <b>්</b> ෂයි

Example C5

<b>60</b> ·	Direct Special B	lack axn		48
	PGmBB	•		98
٠	Dipropylene glyco	ol monomethyl	ether	128

## EP 0 820 B20 A2

2-Mathyl-2,4-pentanediol	<b>5</b> & ·
1,2-propensdiol	<b>4</b> 8
N-Methylpyrrolidone	_ 58
Water	Balance

## Example C8

10	C.I. Readive Red 6	4%
	PGmBE	9%
•	DPGmBE	5%
15	Diathylena glycol mono-n-butyl ether	15%
	Neopantyl glycol	2%
*	1,3-Butanodial	2%
20	N-Acylmathylizurina codium	1.5%
	Water	Balanco

## Example C7

	C.I. Be∋ic Yollow 11	£%3
	PGm8E  Tristhylene glycol monobutyl ether  Glycerin  Trimethylol propane  Trimethylol cihane  Polypropylene glycol having a number average molecular weight of 400  Surfynol 465	10%
20	Tristhylene glycal monobutyl ether	12%
	Glycertn	10%
•	Trimethyld propane	<b>3</b> %
25	Trimethyld etheno	155
	Polypropylene glycal having a number average malecular weight of 400	15%
	Surfynol 465	195
40	Surfynol 104	0.5%
	Water	පිත්රා <b>ර</b> ා

### EP 0 640 600 A2

## Example C6

C.I. Direct Green 1	3%
C.I. Direct Grean 28	1%
PGmBE	10%
Diathylena glycol mono-t-butyl ether	5%
Diathylena glycol mono-n-butyl ether	5%
Diathylona glycol	10%
Tetrapropylano glycol	2%
1,3-Propanedial	5%
Water	Balanca

### 20 Comparative Example C1

15

25

**30** 

## C.I. Direct Green 1

3&

Diethylene glycol mono-n-butyl ether	<b>5</b> 8
Ethylene glycol	<b>15</b> %
Potassium hydroxide	0.18
Hater	Balance

## Comparative Example C2

•		
	C.I.Direct Red 227	2.5%
•	Distinylens glycol mono-n-butyl ether	15%
	Diathylena glycol	5%
40	Chycarin	10%
	Water	Batanca

## Comparative Exemple C3

C.I. Acid Red 254	2.5%
Diathylona glycol mono-n-butyl ether	7%
Diathylena glycol	15%
Water	Betanco

## Evaluation Test C

89

The above ink compositions were used to carry out printing on various types of recording paper specified

In Table 3. The prints thus obtained were evaluated in the same manner as was described in Evaluation Test A1.

The results were as given in Table 3.

10

## EP 0 649 893 AZ

Table 3

	<u> </u>	<del></del>								<del></del>		
Test item	Type of paper				Ex.	C	•				in .	
		1	2	3	4	5	б	7	8	1	2	3
÷	CONQUEROR Paper	9	<b>ø</b>	0	0	0	<b>9</b>	<b>®</b>	8	0	<b>Ø</b>	0
	PAVORIT X Paper	0	0	6	0	<b>Ø</b>	0	9	<b>®</b>	Δ	0	0
	MODO COPY Paper	Ø	0	0	0	0	0	8	9	R	Δ	я
	RAPID COPY Paper	0	9	9	9	0	9	<b>8</b>	<b>6</b>	Δ	0	0
	EPSON BPP Paper	0	0	Ð	•	@	9	0	Ø	я	Δ	R
Bleed-	XEROX P Paper	6	0	@	0	6	Ø	0	9	п	Δ	x
	MEROM 4024 Paper	9	0	0	<b>(</b>	0	Ø	0	<b>@</b>	Δ	0	Δ
	XEROX 10 Paper	9	9	•	0	0	9	0	Ø	Δ	▲	Δ
	NEENACH BOND Paper	0	9	0	0	0	9	0	0	я	Δ	Δ
	RICOFY 6200 Paper	0	9	<b>®</b>	0	0	Ð	0	9	Δ	Δ	Δ
	Yamayuri Paper	0	0	0	0	0	0	<u></u>	ø	ж	x	x
	XEROX R Paper	0	@	0	Ø	ဂ	0	0	0	x	н	n
	COMQUEROR Paper	0	0	@	0	6	•	<b>Ø</b>	<b>@</b>	x	Δ	п
	FAVORIT K Paper	0	0	0	0	0	<b>®</b>	9	⊜	Δ	Δ	Δ
	MODO COPY Paper	0	0	0	<b>©</b>	0	0	9	₽	<b>A</b>	Δ	Δ
Peath-	RAPID COPY Paper	Ō	Ф	0	<u> </u>	ூ	<b>9</b>	0	0	ж	Δ	À
ring	XEROX P Paper	@	0	0	•	0	9	ø	0	я	Δ	ж
	XEROX 4024 Paper	9	0	0	0	<b>6</b>	0	0	0	я	д	я
	RICOPY 6200 Paper	©	0	<b>©</b>	<b>ø</b>	<b>@</b>	0	0	0	н	Δ	Δ
	Yamayuri Paper	9	0	0	0	0	0	0	0	ж	x	ж
	XEROX R Paper	0	0	c	<b>9</b>	0	<b>ø</b>	<b>ø</b>	0	x	п	×

## Exemple D

The following ink compositions of Examples D1 to D8 and Comparative Examples D1 to D3 were prepared by the conventional method.

## Exemplo D1

16

15

20

æ

20

**5**5

C.I. Direct Black 19	2%
PGm8E	. 10%
Triothylono glycol monobutyl other	10%
1,4-Butanadio	2.5%
loibenssell.1	2.5%
Thiodiglycol	5%
₩ater	Balenca

## Example D2

C.I. Food Blad	ck 2	2.5%
PGmBE		. 10%
Diathylena gly	yool mono-n-butyl ether	10%
Dipropyleno g	lycol	3%
Thicdiglycol		3%
Urea		5%
Water		contales

## Example D3

C.i. Acid Green 9	2.5%
PGmBE	10%
1-Methyl-1-mathoxy butand	15%
Propylono glycel	<b>2</b> 5%
Thiediglycol	5%
1.5-Pentanedic	5%
Water	Belanco

### EP 0 849 844 AZ

## Example D4

Bayacript Black SP Ilquid 20%

DPGmBE 10%

Propylene glycol monoathyl ethar 10%

Thiodiglycol 12%

Tripropylene glycol 3%

1,3-Dimathyl-2-Imidazolidinona 6%

Water Belonce

Exemple D5

Direct Special Black AXN

PGm3E

Dipropytone glycol monomothyl other

Thicdiglycol

1.2-Propanedial

N-Methylpyrrolidons

5%

Weter

Balanca

Exemplo D8

80

C.I. Reactive Red 6 4% PGmBE 8% 5% DPGm8E Disthylons glycol mono-n-butyl ether 15% 20% Thiodigiyod 1,3-Butancolid 2% N-Acylmathyltaurino codium 1.5% Salanco Weber

## Example D7

	C.I. Basic Yellow 11	5%
<b>5</b>	PGm8E	10%
	Triathylana glycol monobutyl ether	2%
	Glycarin	10%
10	Trimathylol propens	3%
	Trimothytol othera	1%
	Polyethylene glycol having a number average molecular weight of 400	, 1%
15	Thiodiglycol	30%
•	Surfynoi 465	0.5%
	Water	Balanca

20

## Example D8

	C.I. Direct Green 1	38
245	C.I. Direct Green 28	18
	PGmBE	103
20	Diethylene glycol mono-t-butyl ether	5%
	Diethylene glycol mono-n-butyl ether	5%
	Diethylene glycol	10%
	Thiodiglycol	0.5%

29

# 1,3-Propanediol

. Water

50 Balance

# Comparative Example D1

	1
C.I. Direct Green 1	3%
Distinylans glycol mono-n-butyl ether	5%
Ethylene glycol	15%
Potestum hydroxids	0.1%
Water .	Balanca

**£0** 

### Comparative Example D2

C.I.Direct Elack 154	2.5%
Diathylens glycol mono-n-butyl ether	16%
Diathylena glycol	5%
Glycerin	10%
Water	Balanca

## Comparative Exemple D3

C.I. Acid Red 254	2.5%
Diathylena glycol mono-n-butyl ether	7%
Diathylena glycol	15%
Water	Balanca

## Evaluation Test D1

The above ink compositions were used to carry out printing on various types of recording paper specified in Table 4. The prints thus obtained were evaluated in the same manner as was described in Evaluation Test A1.

The results were as given in Table 4.

Table 4

Test item	Type of paper				Ex.	D					gmc I.x	
		1	2	3	4	5	6	7	8	1	2	3
	COMQUEROR Paper	9	0	<b>®</b>	<b>9</b>	0	0	0	<b>6</b>	0	0	C
	FAVORIT E Paper	0	0	<b>®</b>	0	<b>9</b>	<b>9</b>	<b>3</b>	<b>9</b>	Δ	0	C
	MODO COPY Paper	0	0	0	0	0	<b>©</b>	<b>@</b>	<b>9</b>	Ħ	A	R
	RAPID COPY Faper	0	<b>6</b>	0	6	9	0	0	9	<u>A</u>	Ó	~
	EPSON EPP Paper	0	6	6	0	0	0	0	0	×	Δ	s
Bleed-	XEROX P Paper	0	<b>®</b>	0	0	0	0	0	<b>©</b>	ж	Δ	]
ing	XEROX 4024 Paper	0	<b>9</b>	<b>6</b>	0	0	0	0	0	Δ	0	<u> </u>
	KEROX 10 Paper	0	<b>ø</b>	0	0	0	0	0	<b>6</b>	Δ	Δ	4
	NEENACH BOND Paper	Ø	0	0	0	0	0	0	0	я	Δ	L
	RICOPY 6200 Paper	0	0	@	0	0	0	0	@	Δ	Δ	L
	Yamayuri Raper	0	0	0	0	0	0	0	0	я	n	
	KEROK R Paper	0	9	Ö	0	0	0	0	0	×	x	
	COMQUEROR Paper	9	0	0	0	0	9	6	0	ж	Δ	Ĺ
	FAVORIT X Paper	9	0	0	0	0	0	0	0	Δ	A	<u> </u>
	MODO COPY Paper	Ø	0	0	9	0	0	Ø	100	Δ	Δ	L
	RAPID COPY Paper	9	<b>@</b>	0	0	<b>(</b>	6	<b>6</b>	9	x	Δ	L
reath- ring	XEROX P Paper	· @	@	0	0	0	0	0	0	x	Δ	L
	XEROX 4024 Paper	0	0	0	0	0	0	0	6	ж	ж	L
	RICOPY 6200 Paper	<b>©</b>	0	0	: @	0	0	0	0	я	Δ	
I	Yamayuri Paper	0	0	0	0	0	0	0	0	ж	,ж	
	XEROX R Paper	9	6	C	@	0	0	0	0	ж.	ж	

**5**5

### EP 0 840 800 A2

## Evaluation Test D2

In the int composition of Example D1, the thiodiglycol concentration was varied. A change in the total amount of the composition caused by the increase or decrease in the amount of thiodiglycol add was belanced by water. The intermittent printing time for the inter was measured. The results were as shown in Fig. 4.

### Exemplo E

The following ink compositions of Examples E1 to E8 and Compositive Examples E1 to E3 were prepared by the conventional method.

The fluorosurfactant used in the following examples and comparative examples was Flargent 251 (manufactured by Neos Co., Ltd.).

### Exemple E1

15

C.I. Direct Black 19	2%
PGm8E	10%
Triothylono glycal manabulyl other	10%
1.4-Butanadiol	2.5%
1,8-Hexanediol	2.5%
Fluorocuriaciant	0.02%
Water .	<b>Belenc</b> o

#### Exemplo E2

C.I. Food black 2	2.5%
PGm8E	10%
Distinyons glycol mono-n-butyl ether	10%
Digrapyiene giyeed	3%
Ruoresurfectent	0.03%
Urea	5%
Water	8atance

## Example E3

	C.I. Acid Green 9	2.5%
	PGmBE	10%
50	1-Methyl-1-methoxy butanol	15%
	Propylene glycol	5%

## Fluorosurfactant 1,5-Pentamediol Water

0.1% 5% Salance

## Example E4

10

20

	Bayacript Black SP liquid	20%
	DPGm8E	10%
•	Propylene glycol monoethyl ether	10%
	Fluorosurfactant	0.2%
	Tripropylene glycol	<b>3</b> %
•	1,3-Diməthyi-2-imidazolidinone	5%
	Water :	පින්නඟය

## Example E5

Direct Speedal Black AXW	4%
PGmBE	9%
Dipropylona glycol monomethyl ether	12%
Fluorosurfactant	1 PPM
1,2-Propanadio	· 4%
N-Methylpyrrolidona	5%
Wolcz	ම්ක් <b>න</b> ාලය

## Example E8

•	C.I. Residive Red 8	4%
	PGmBE	9%
	DPGm8E	5%
	Disthylone glycol mono-n-butyl ether	16%
	Fluorosurisciant	1.0%
	1,3-Butancolid	2%
	N-Acylmathylizurina sodium	1.5%
	Water	Balanca

## Example E7

	C.I. Basic Yellow 11	5₺
5	PGmBE	10%
	Triethylene glycol monobutyl ether	28
	Glycerin	10%
	Trimethylol propane	3₺
10	Trimethylol ethane	18
	Polyethylene glycol having a number	•
	average molecular weight of 400	18
15		
,	Fluorogurfactant	0.38
••	Surfynol 465	0.58
20	Hator	Balance

## Example E8

<b>26</b>	C.I. Direct Green 1	3%
	C.I. Diraci Green 28	1%
	PGmBE	10%
<b>20</b> ·	Diathylena glycol mono-t-butyl ether	5%
	Distinyions glycol mono-n-butyl ether	5%
•	Diathylona glycci	10%
<b>35</b>	Fluorosuriectent	0.5%
·	1,3-Propanedial	5%
•	Water	Batance

## Comparative Example E1

_	C.I. Direct Green 1	3%
<b>45</b>	Diathylena glycol mono-n-butyl ethar	5%
	Ethylene glycol	15%
•	Potencium hydroxida	0.1%
<b>£0</b>	<del>W</del> ater	පිවසාල

65

## Comparative Example E2

C.I. Direct Red 164	2.5%
Distinyions glycal mano-n-butyl cities	16%
Diathylona glycol	5%
Glycarin	10%
Water	Balanca

## Comparative Example E3

15	C.I. Actd Red 254	2.5%
	Diathylena glycol mono-n-butyl ether	798
	Diathylena glycol	15%
20	Water	Balanca

## Evaluation Test E

22

45

The above ink compositions were used to carry out printing an various types of recording paper specified in Table 5. The prints thus obtained were evaluated in the same manner as was described in Evaluation Test A1.

The results were as given in Table 5.

## EP 0 840 840 AZ

Table 5

Test itom	Type of pager	Ex. E								Comp. En. E		
		1	2	3	4	5	6	7	8	1	2	3
	COMQUEROR Paper	0	0	9	0	<b>3</b>	<b>ø</b>	0	<b>®</b>	٥	0	0
	FAVORIT X Paper	0	<b>©</b>	0	   (0	<b>9</b>	0	Ø	(a)	Δ	0	0
	WODO COPY Paper	0	Ø	0	0	0	<b>Ø</b>	<b>Ø</b>	•	ĸ	Δ	×
	RAPID COPY Paper	6	<b>9</b>	0	Ð	<b>®</b>	<b>3</b>	<b>®</b>	€	Δ	0	0
	EPSON EPP Paper	Œ	<b>9</b>	<b>@</b>	0	න	0	<b>@</b>	@	n	Δ	я
Bleed-	XERUX P Paper	<b>©</b>	0	@	0	0	<b>©</b>	0	<b>9</b>	X	Δ.	я
ing	XEROX 4024 Paper	0	❷	0	0	0	0	<u></u>	<b>(a)</b>	Δ	0	Δ
	XEROX 10 Paper	0	0	0	@	0	Ð	<b>Ø</b>	0	Δ	Δ	Δ
	NEENACH BOND Paper	9	0	0	0	0	Ð	0	0	. π	Δ	▲
	RICOPY 6200 Paper	9	9	0	0	0	0	9	0	Δ	Δ	Δ
	Yamayuri Raper	0	0	0	0	0	0	0	0	и	я	я
	KEROX R Paper	0	<b>@</b>	0	0	0	<b>ø</b>	0	0	ĸ	×	я
:	COMQUEROR Paper	0	8	9	0	0	9	0	0	×	Δ	×
	FAVORIT X Paper	9	6	0	0	0	0	9	<b></b>	Δ	Δ	Δ
	MODO COPY Paper	0	6	Ø	0	<u></u>	0	<u> </u>	<b>ø</b>	A	Δ	Δ
,	RAPID COPY Paper	<u></u>	0	0	0	0	0	0	0	н	▲	Δ
Feath- ring	XEROX P Paper	@	0	9	0	0	0	0	0	я	Δ	Ħ
[	XEROX 4024 Paper	@	0	0	0	0	<b>9</b>	0	0	и	х	ж
	RICOPY 6200 Paper	@	0	0	0	0	9	ø	6	ж	Δ	Δ
	Yamayuri Paper	0	0	0	0	0	0	0	0	ж	ж	ĸ
	XEROX R Paper	0	0	0	0	0	9	Ø	0	×	ж	ĸ

ก

\_\_\_

#### 57 0 649 830 A2

#### Claims

- 1. An ink composition for ink jet recording, comprising a dye, propylene glycol mono-n-butyl either (PGmBE) and/or dipropylene glycol mono-n-butyl either (DPGmBE) and a water-coluble glycol either other than PGmBE and DPGmBE, the total amount of PGmBE and DPGmBE being 3 to 30% by weight based on the ink composition.
- 2. The ink composition eccording to claim 1, wherein the total amount of PGmBE and DPGmBE is 3 to 10% by weight based on the ink composition.
- The ink composition according to claim 1, wherein the water-coluble glycal ether is extected from the group consisting of ethylene glycal monosikyl ethers, diethylene glycal monosikyl ethers, triethylene glycal monosikyl ethers, propylene glycal monosikyl ethers, dipropylene glycal monosikyl ethers and sikoxycubalituted allphatic sicahols.
- 4. The ink composition according to claim 1, wherein the water-coluble glycel other is selected from the group consisting of ethylene glycel mono-C<sub>1-0</sub>alkyl ethers, tribinylene glycel mono-C<sub>1-0</sub>alkyl ethers, propylene glycel mono-C<sub>1-c</sub>alkyl ethers, dipropylene glycel mono-C<sub>1-c</sub>alkyl ethers and C<sub>1-calk</sub>eny-substituted C<sub>1-calk</sub>eny-substitut
- 5. The interposition according to claim 1, wherein the ratio of the water-soluble glycol either to PGmBE and DPGmBE is not less than 0.5.
  - 6. The intercomposition according to claim 1, wherein the amount of the water-soluble glycol ether is 5 to 60% by weight based on the intercomposition.
  - 7. The introcomposition according to claim 1, which further comprises urea and/or a urea derivative.
  - The ink composition according to claim 7, wherein eald urea derivative is ethylensurea, thiourea, bluret or tetramethylurea.
  - 9. The link composition according to claim 7, wherein the amount of the urea and/or urea derivative is 2 to 20% by weight based on the link composition.
    - 10. The ink composition according to claim 1, which further comprises thirdighypol.
  - 11. The link composition according to claim 10, wherein the amount of the thicdiglycal is 0.5 to 30% by weight based on the ink composition.
    - 12. The ink composition according to claim 1, which further comprises a surfactant.
- 49 19. The introcomposition according to claim 12, wherein the surfactant is a fluorecurfactant.
  - 14. The ink composition according to claim 13, wherein the concentration of cald fluorecurfactant to 1 to 10,000 ppm.
- 45 15. The ink composition according to claim 13, wherein the fluorecurfoctant is an emphasized surfactant or a monitoric surfactant.
  - 10. The introoperation according to claim 1, wherein the formability of said introoperation as measured by the Ross-Miles method is not more than 200 mm as the initial value and not more than 100 mm 5 min after the initiation of the test.
    - .17. The introcomposition eccording to claim 1, which has a surface tancion of 15 to 40 mN/m.
  - 18. The ink-composition according to claim 1, wherein the forward contact angle of eald ink composition with a nexalle plate of an ink jet recording head to 10 to 50°.
  - 10. Use of an ink composition comprising a dye, propylene glycol mono-n-butyl either (PGm3E) and/or dipropylene glycol mono-n-butyl either (DPGm3E) and a water-coluble glycol either other than PGm3E and

10

15

DPGmBE, the total amount of PGmBE and DPGmBE being 3 to 30% by weight based on the ink composition, for ink jet recording.

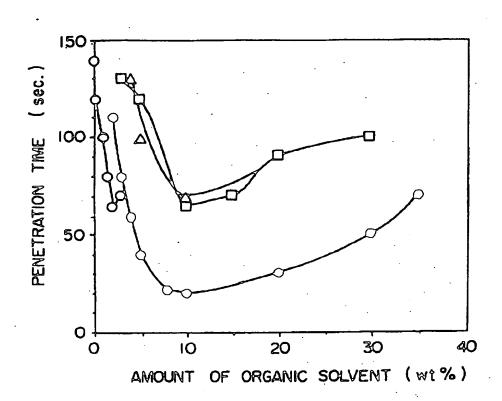


FIG. I

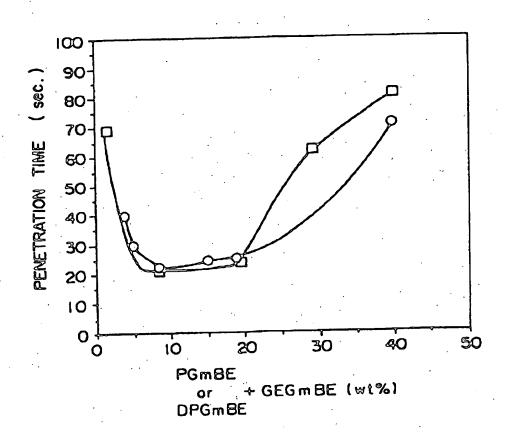


FIG. 2

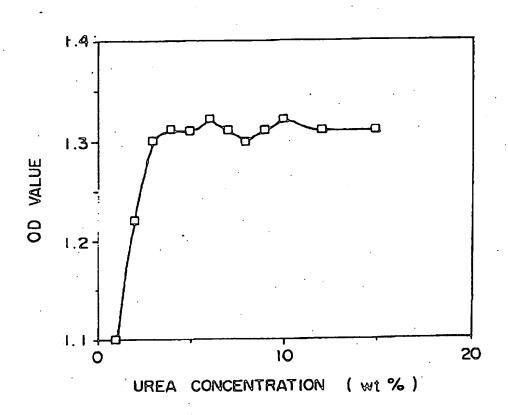


FIG. 3

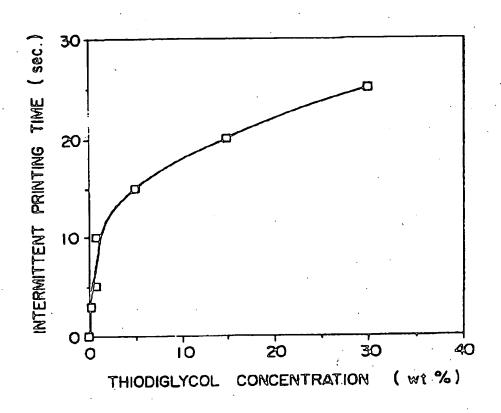


FIG. 4